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IDENTIFICATION OF THE MINERALS FROM HALL'S GAP, KENTUCKY, BY X-RAY ANALYSIS

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ABSTRACT:

Hall's Gap, Kentucky, well known to mineral collectors for fine millerite and other minerals, has, prior to this date, never has had the identities of its minerals established by any method other than visual determination. This report provides positive identities for the various minerals by way of an X-ray diffractometer which was used to determine the characteristic diffraction patterns for the minerals undergoing analysis. The data obtained largely confirmed earlier observations. The exception was the solid nodules which were previously thought to be composed of marcasite. Diffraction patterns obtained, however, placed the nodules into two groups, one of which was those consisting of sulfides other than marcasite while the second group was composed of phosphates. In addition, an attempt to obtain a diffraction pattern for the controversial honessite was unsuccessful and the status of the mineral remains uncertain. Excellent color illustrations provide the reader with some fine examples of the various minerals found in the area.

INTRODUCTION:

Hall's Gap, Kentucky, has long been a well known source of fine specimens of millerite, accompanied by other sulfides, calcite, dolomite, and quartz, all contained in geodes. The occurrence is located 43 miles south of Lexington, Kentucky, in a roadcut on U.S. 27 at the town of Hall's Gap. The cut is located 1000 feet north

of the intersection of state route 1247 with that highway.

The productive area is confined to the Wildie Member of the Borden Formation and is Mississippian in age. The geodes occur in a massive, greenish-gray siltstone approximately 4 feet thick. Shaly glauconite beds about 12 inches thick above and below the bed facilitate recognition.

Until this time, all identification of the minerals found in the geodes has been based on visual determination. This report contains positive identification of the minerals by means of X-ray diffraction analysis which confirms, and in some cases, modifies the earlier visual identifications. In addition to the work on the geodes, a diffraction pattern of the siltstone matrix in which the geodes occur was obtained.

X-RAY DIFFRACTION ANALYSIS:

The instrument used was a General Electric, Model XRD-6, diffractometer equipped with a Cu-target X-ray tube. It was operated with the settings listed below:

1. Cu K-alpha X-rays, Ni filtered.
2. 45KVP, 15 m.a.
3. 3° Soller slit.
4. DELTA-E in. (Pulse height discrimination)

Approximately five dozen geodes were collected from the roadcut and carefully examined to provide as complete a suite as possible of the minerals present. After being

powdered and mounted on glass slides, the mineral samples were scanned of the diffractometer for values of 2θ from 5° through 70°. The sphalerite and the siltstone matrix were scanned through 80° in an attempt to gain additional peaks on the strip charts. The observed diffraction patterns and the ASTM diffraction patterns for the minerals with which they were matched are presented in the following tables:

Sample # 1: (Siltstone matrix)

QUARTZ

2θ-Chart	2θ-ASTM
26.70	26.66
20.89	20.85
50.18	50.21
36.55	36.56
39.50	39.49
68.23	68.20
42.50	42.48
68.38	68.37

ILLITE

2θ-Chart	2θ-ASTM
8.83	8.84
19.84	19.82
26.70	26.77
34.36	34.36
60.50	60.51
-----	37.15
55.18	55.09
17.88	17.92

ALBITE

2θ-Chart	2θ-ASTM
28.08	28.10
23.70	23.71
27.84	27.78
22.00	22.00
22.91?	22.91
-----	24.46
28.53	28.53
30.54	30.54

MUSCOVITE

2θ-Chart	2θ-ASTM
26.80	26.85
8.89	8.89
34.98	34.97
45.58	45.51
29.90	29.91
17.88	17.85
-----	81.67
27.84	27.89

GLAUCONITE

2θ- Chart	2θ-ASTM
8.75	8.75
34.70	34.67
19.60	19.60
26.70	26.77
-----	37.54
61.35	61.35
24.54	24.52
-----	28.89

Sample # 2: (Solid nodule, sulfide type)

QUARTZ

2θ-Chart	2θ-ASTM
26.66	26.66
20.80	20.85
50.20	50.21
36.55	36.56
-----	39.49
-----	68.20
42.46	42.48
-----	68.37

PYRITE

2θ-Chart	2θ-ASTM
56.36	56.34
33.06	33.07
37.10	37.10
40.76	40.79
47.44	47.45
28.54	28.54
-----	95.31
-----	84.64

ARSENOPYRITE

2θ-Chart	2θ-ASTM
37.00	36.99
33.69	33.69
50.14	50.12
56.24	56.30
69.70?	69.76
-----	24.39
-----	31.85
-----	51.95

SPHALERITE

2θ-Chart	2θ-ASTM
28.54	28.58
47.52	47.56
56.36	56.34
33.10	33.12
-----	76.88
-----	88.68
69.56	69.59
-----	95.68

Sample # 3: (Solid nodule,phosphatic type)

QUARTZ

2θ-Chart	2θ-ASTM
26.68	26.66
20.85	20.85
50.21	50.21
35.56?	36.56
39.43	39.49
-----	68.20
42.48	42.48
-----	68.37

(SYN)
FLUORAPATITE

2θ-Chart	2θ-ASTM
31.92	31.96
33.15	33.15
32.29	32.29
-----	36.80
34.17	34.17
49.63	49.63
46.92	46.90
40.08	40.07

(SYN)
CHLORAPATITE

2θ-Chart	2θ-ASTM
32.21	32.21
31.80	31.80
32.98	32.93
25.90	25.90
49.52	49.51
46.75?	46.75
34.10	34.08
39.85	39.85

(SYN)
HYDROXYLAPATITE

2θ-Chart	2θ-ASTM
31.80	31.80
32.21	32.21
39.85	39.83
25.90	25.90
49.52	49.51
46.75?	46.75
34.10	34.08
39.85	39.85

Sample # 4:

MILLERITE

2θ-Chart	2θ-ASTM
32.28	32.23
48.92	48.89
35.70	35.73
18.48	18.45
40.58	40.59
50.18	50.18
-----	30.34
52.72	52.69

Sample # 5: ALTERED MILLERITE ("HONESSITE")
Negative results; sample gave diffraction pattern identical to that of millerite. (See above)

Sample # 6:

PYRITE

2θ-Chart	2θ-ASTM
56.30	56.34
32.95	33.07
37.00	37.10
40.64	40.79
47.33	47.45
28.50	28.54
-----	95.31
-----	84.64

Sample # 7:

CHALCOPYRITE

2θ-Chart	2θ-ASTM
29.46	29.48
49.16	49.14
57.96	57.96
-----	91.42
48.84	48.83
-----	92.30
58.76	58.69
-----	98.45

Sample # 8:

SPHALERITE

2θ-Chart	2θ-ASTM
28.63	28.58
47.58	47.56
56.42	56.34
33.14	33.12
76.80	76.88
-----	88.68
69.52	69.59
-----	95.68

Sample # 9:

DOLOMITE
(FERROAN)

2θ-Chart	2θ-ASTM
30.90	30.84
40.94	41.04
50.34	50.36
50.96	50.96
-----	24.05
-----	33.37
37.20	37.29
44.77	44.87

Sample # 10:

CALCITE

2θ-Chart	2θ-ASTM
29.52	29.43
39.50	39.43
43.30	43.18
47.65	47.53
48.65	48.55
36.08	36.00
23.12	23.04
57.54	57.45

Sample # 11:

QUARTZ

2θ-Chart	2θ-ASTM
26.58	26.66
20.72	20.85
50.15	50.21
36.44	36.56
39.44	39.49
68.20	68.20
42.36	42.48
68.37	68.37

Sample # 12:

CHALCEDONY
(QUARTZ)

2θ-Chart	2θ-ASTM
26.66	26.66
20.88	20.85
50.20	50.21
36.62	36.56
39.50	39.49
68.24	68.20
42.50	42.48
68.42	68.37

Prior to the scanning of each of the preceeding samples, tentative identifications were made on the basis of directly observable characteristics such as color, luster and habit. In other words, the specimens were identified as they had been previously, according to visual criteria. The diffraction pattern from the test sample in each scan was then checked against the known diffraction pattern for the mineral that the sample was assumed to be. Results confirmed the accuracy of the previous identifications for all of the samples except the solid nodules and the altered millerite.

It had been assumed that all of the solid nodules from the area were composed of marcasite. Sample # 2 was found to contain quartz, pyrite, arsenopyrite, and sphalerite, but no marcasite. Sample # 3 produced some unexpected results. It was composed of quartz, fluorapatite, chlorapatite, and hydroxylapatite. Prior to this analysis, the presence of phosphatic nodules in the area had not been considered.

The green alteration product of millerite presented a different problem. The initial fear that the actual quantity of millerite altered would be insufficient to yield results in the diffractometer was confirmed, when the results showed a pattern which was a duplicate for that of millerite.

This alteration product, known as honessite, was first found near Milwaukee, Wisconsin prior to 1959 and

shortly afterward at Hall's Gap. Its status as a mineral is not clear and little seems to be known about it. Heyl, Milton and Axelrod, writing in AMERICAN MINERALOGIST, state: " The chemical literature.....lists many syntheses of basic nickel sulfate. It is not possible, however, to identify either these sulfates or the wisconsin substance precisely enough to say that any one of them has a particular formula. Attempts to synthesize a compound having properties like those of the Wisconsin mineral were unsuccessful."

For reasons unknown, the name honessite was approved by the I.M.A.. While it is not recognized as a mineral by Strunz (MINERALOGISCHE TABELLEN, 1970), it is accorded full status by the usually conservative Hey (APPENDIX TO THE CHEMICAL INDEX OF MINERALS, 1963). Significantly, there is no reference to the material in the A.S.T.M. POWDER DIFFRACTION DATA FILE. In spite of the controversy which surrounds it, this much is certain; the Hall's Gap material is formed through the alteration of and is pseudomorphous after millerite.

X-RAY FLUORESCENCE ANALYSIS:

Finally, one of the solid nodules (Sample # 2) was subjected to a scan by the X-ray fluorescence spectrometer. The instrument used was a G.E. Model XRD-6 diffractometer equipped with a Mo-target X-ray tube and a LiF (220) crystal. It was operated with the settings listed below:

1. 65 KVP, 55m.a.
2. E = 3.5V, DELTA-E = 15V
3. 0.010 Soller slit
4. Peaked on Fe K-ALPHA

The nodule was pulverized and sifted through a 200 mesh screen, after which it was compressed into a pellet for analysis. The scan was run for values of 2θ from 55° through 96° in order to determine if zinc, copper, nickel, cobalt, iron or manganese were present. Iron, copper, and cobalt were found to be present. Manganese, zinc and nickel did not register on the chart.

PHOTOGRAPHY OF THE MINERAL SPECIMENS:

It was decided that several good photographs of representative specimens from the Hall's Gap area would useful. A Miranda DR 1.9 single lens reflex camera was used in conjunction with a bellows extension and a lens with a focal length of 5 centimeters. Film used was Kodak High Speed Ektachrome (Tungsten), exposed and processed for ASA's of 125 and320. A light source with a filament temperature of 3200°K (The film used was balanced for this light source.) to reduce the filters required to one color correction type filter (CC10R) for exposures exceeding 1/10 second. Due to the small areas being photographed and the relatively long exposures required, a highly accurate CdS spot type exposure meter with a field of view sensitive to light covering 2° of arc was employed.

Specimens to be photographed were selected with the

intention of obtaining the best quality and greatest number of euhedral specimens possible while keeping the actual number of photographs at a reasonable figure.

In addition, it is felt that these illustrations will provide the reader with useful information about the mineral associations and their orders of occurrence in a far more attractive manner than by the use of mere words. The illustrations will be found on pages 18 through 22.

Readers further interested in the material from Hall's Gap are referred to the article Mineral Rings and Cylinders , found in MINERALOGICAL RECORD, Vol. 1, # 3, for some spectacular micrographs of pyrite from the area in the form of rings.

ACKNOWLEDGMENTS:

Thanks are due at this point to Dr. Faure for freely placing at my disposal his time, knowledge, and facilities, which made this paper possible.

FIGURE # 1: An outstanding millerite bearing geode in matrix. Diffraction data for the identification of millerite was obtained with similar material. The exposed area of the geode is about the size of a tewnty-five cent piece.

FIGURE # 2: "Honessite", the controversial alteration product of millerite. Note the zig-zag shaped pyrite in the center of the geode. This specimen is approximately two inches long on its long axis.

FIGURE # 3: Typical calcite from Hall's Gap in the form of rosettes and skewed rhombohedrons. Note the euhedral pyrites which formed prior to the calcite. This specimen is roughly two inches in diameter.

FIGURE # 4: A geode bearing pyrite, chalcopyrite, and millerite. This specimen is about the diameter of a five cent piece.

FIGURE # 5: A typical millerite-pyrite association on a base of micro-crystalline quartz. The pyrite has grown around the crystals of millerite. Specimen is is one inch long on its long axis.

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